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Advanced Diamond Anvil Techniques (Customized Diamond Anvils)

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February 13, 2009

Advances in Customized Diamond Anvils
Skye, United Kingdom
May 26, 2008 through June 6, 2008

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Advanced Diamond Anvil Techniques

(Customized Diamond Anvils)

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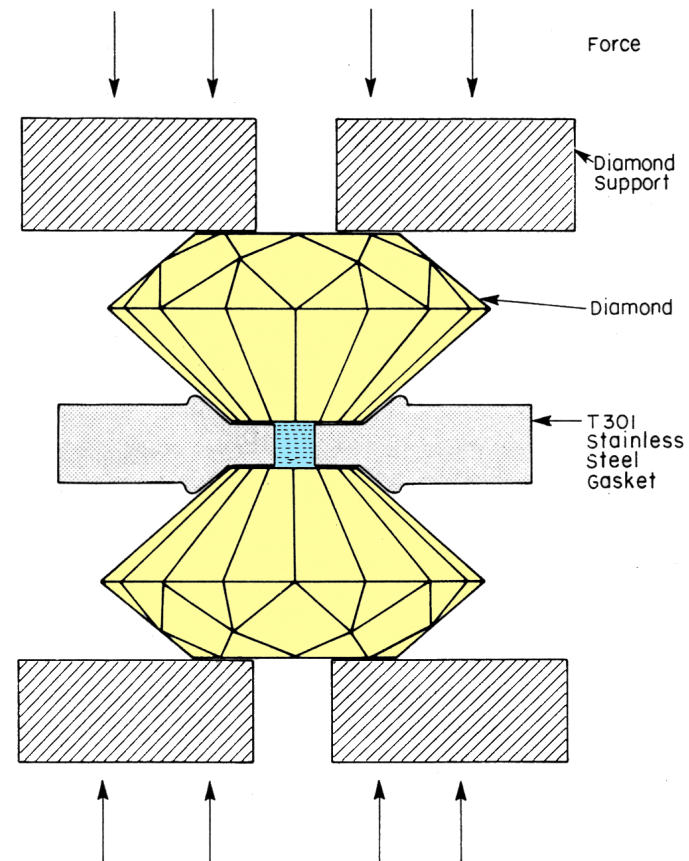
LLNL-PROC-410545

This work performed under the auspices of the U.S. Department of Energy by
Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344

Desirability of Customizing Diamond Anvils for Certain Tasks

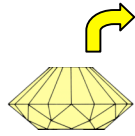
Can the diamond anvils themselves be modified for various types of experiments?:

- Electrical Transport Properties
(Conductivity, Hall effect)
- Magnetic Susceptibility
- Thermal Conductivity
- Thermoelectric Power
- Specific Heats
- Dielectric Properties
- Experiments at High-P/High-T
-



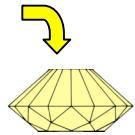
Fabrication Tools that can be applied to Diamond

Diamond Removal:



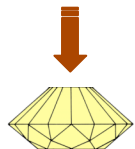
- Polishing
- Laser Drilling
- Plasma Etching

Diamond Growth:



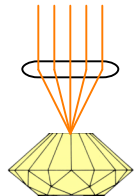
- Chemical Vapor Deposition (CVD)
(low-pressure plasma)

Metal Deposition:



- Sputtering
- e-Beam Evaporation
- Focused Ion Beam (FIB) deposition

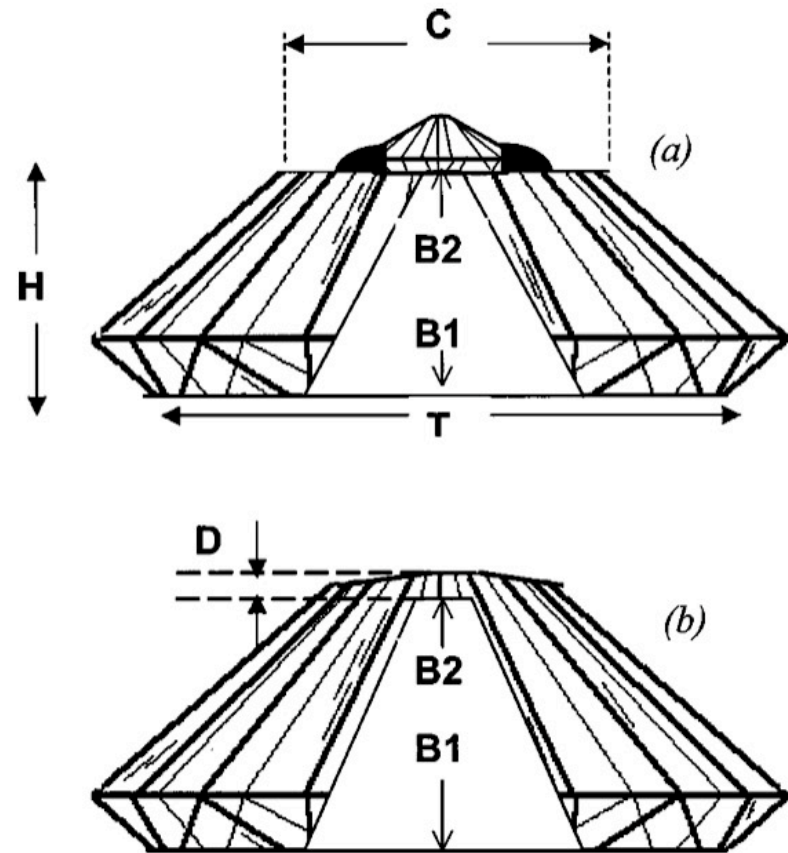
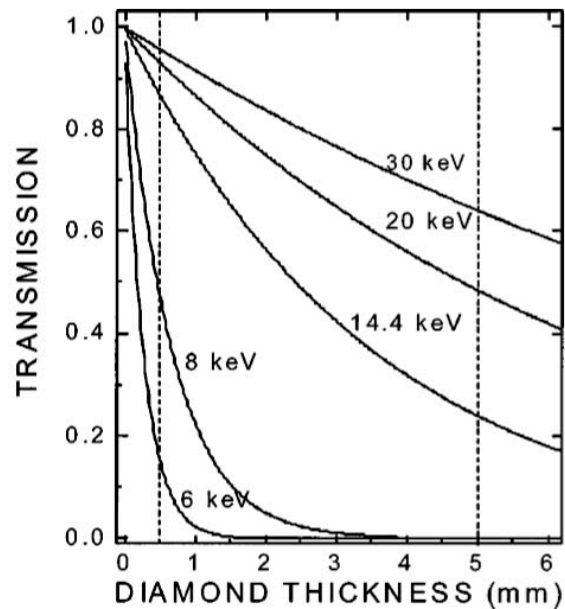
Lithography: (patterning)



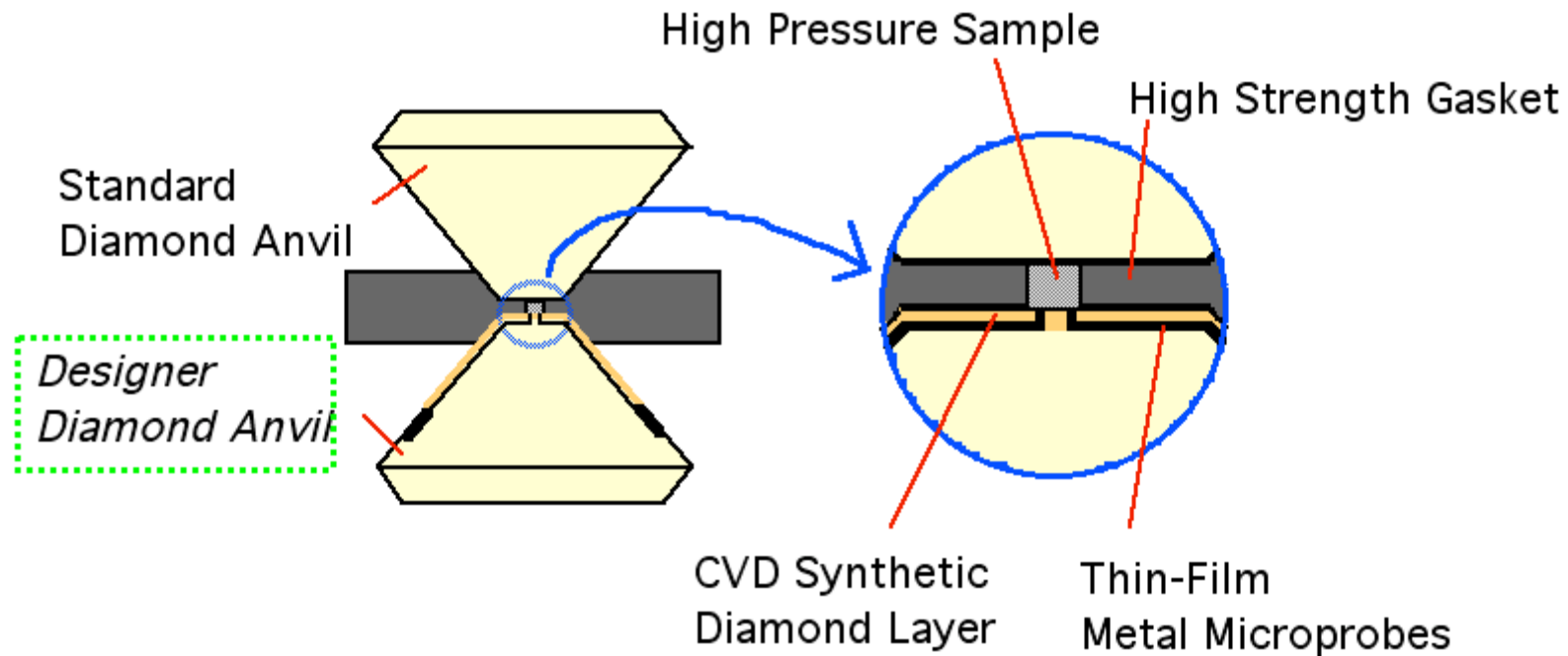
- Projection lithography (2D)
- Laser Pantography (3D)

Example: Laser-Drilled Diamond Anvils for Low Energy X-Rays

- Compound diamond anvil consisting of a miniature diamond anvil combined with a laser drilled diamond anvil
- Diamond thickness reduced from ≈ 5 mm to <1 mm for better transmission of low-energy x-rays.



“Designer” Diamond Anvils



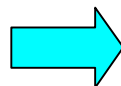
Fabrication Tools:

- Thin-film metal deposition
- Lithographic patterning (both 2D and 3D)
- High-quality, *epitaxial* diamond deposition

Fabrication of a Designer Diamond Anvil

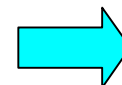
- **Lithographic Fabrication**

(2D & 3D
Lithography)

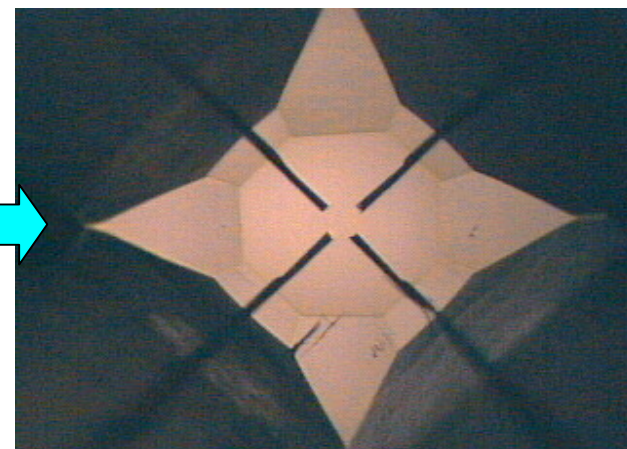
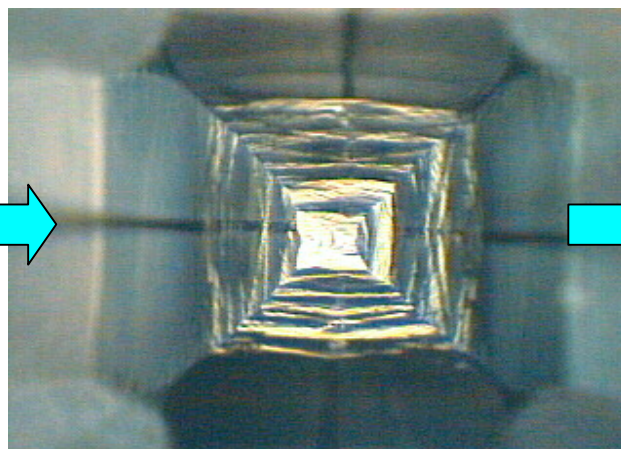
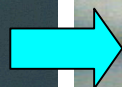
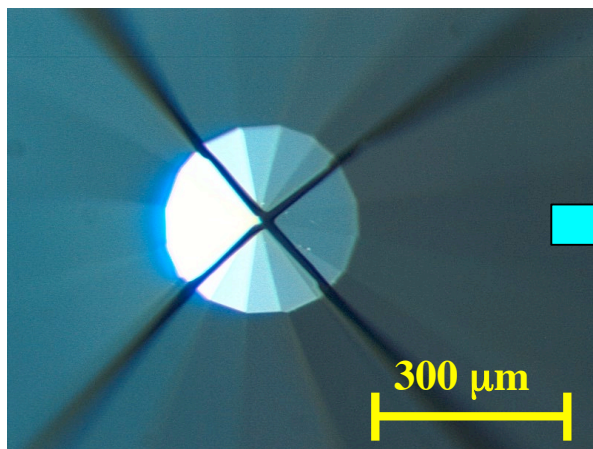


- **Diamond Deposition**

(UAB epitaxial
process)



- **Final Polishing**



Metal Deposition onto Diamond

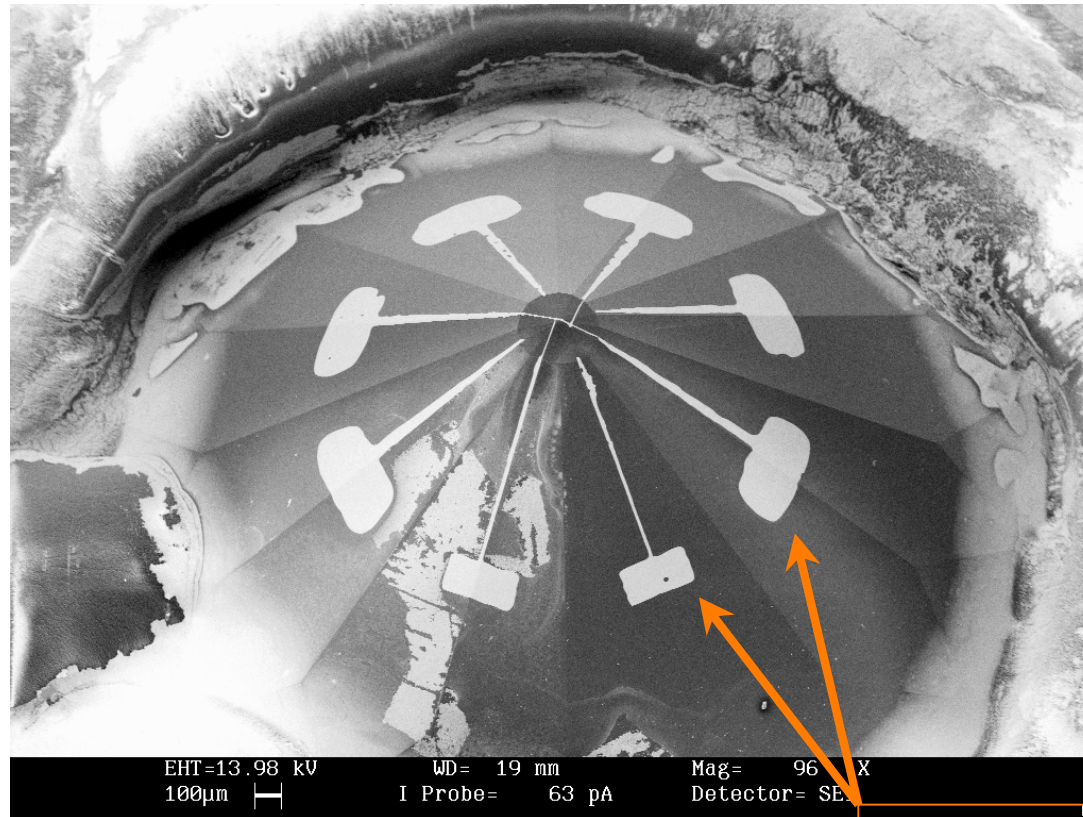
- Diamond is an extremely non-stick surface!
 - > diamond surface is naturally terminated by a monolayer of oxygen or hydrogen.
 - > must strip away this monolayer immediately before deposition.

Successfully depositing well adhering metal films to diamond requires (in our experience):

- Sputter deposition (as opposed to e-beam evaporation)
- A very clean vacuum system
- A pre-etching step in which the diamond surface is etched with argon immediately before sputtering
- Using a carbide-forming metal
(e.g., W, Zr, Mo adhere well to diamond. Au and Cu do not.)

Electrical Probe Pattern on a Diamond Anvil

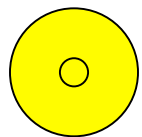
- 0.5 micron thick tungsten film
- Lithography used to pattern the probes



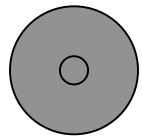
Electrical
Contact Pads

Lithography on Diamond Anvils

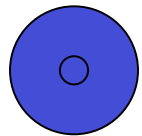
Top View
of a diamond anvil



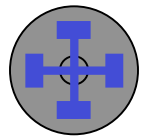
Starting anvil



Sputter deposition of a metal film ($\approx 0.5 - 1.0 \mu\text{m}$ thick)



Electrodeposit photoresist onto metal film



Laser Pantography

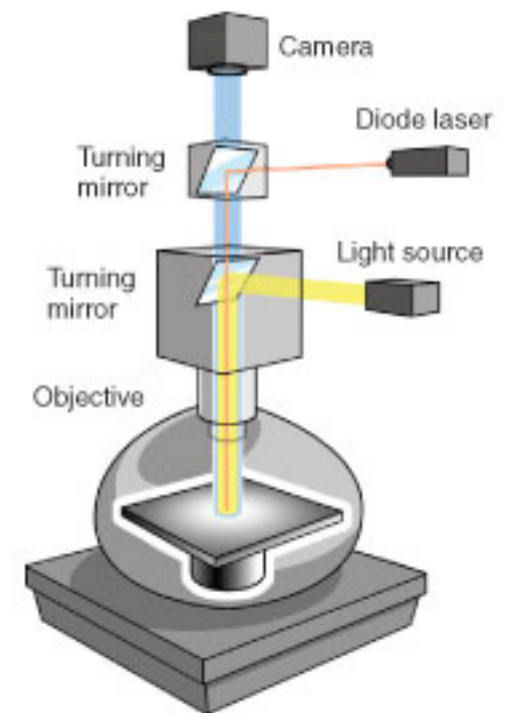


Acid etch all the exposed metal

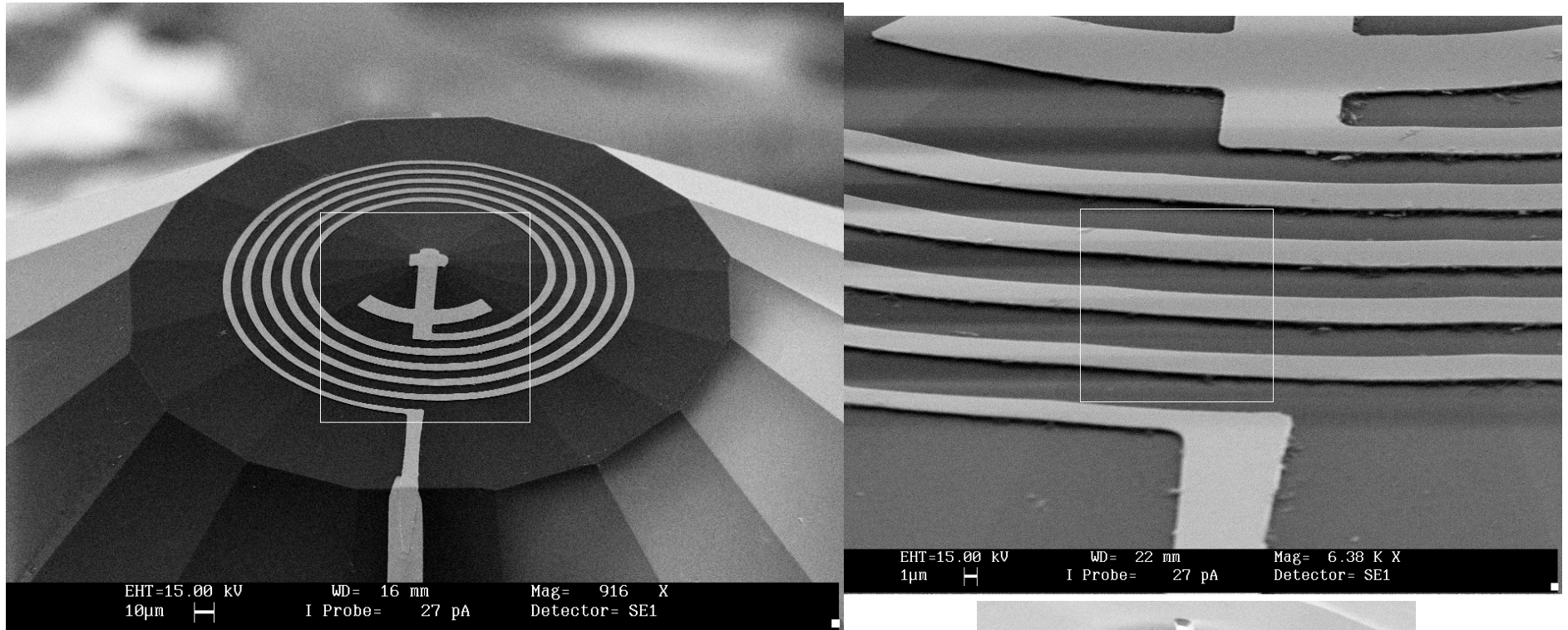


Dissolve away the remaining photoresist

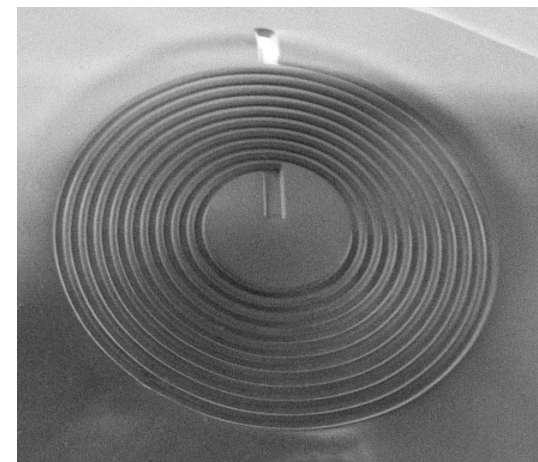
3D Laser Pantography System



Standard 2D Projection Lithography used for the Culet

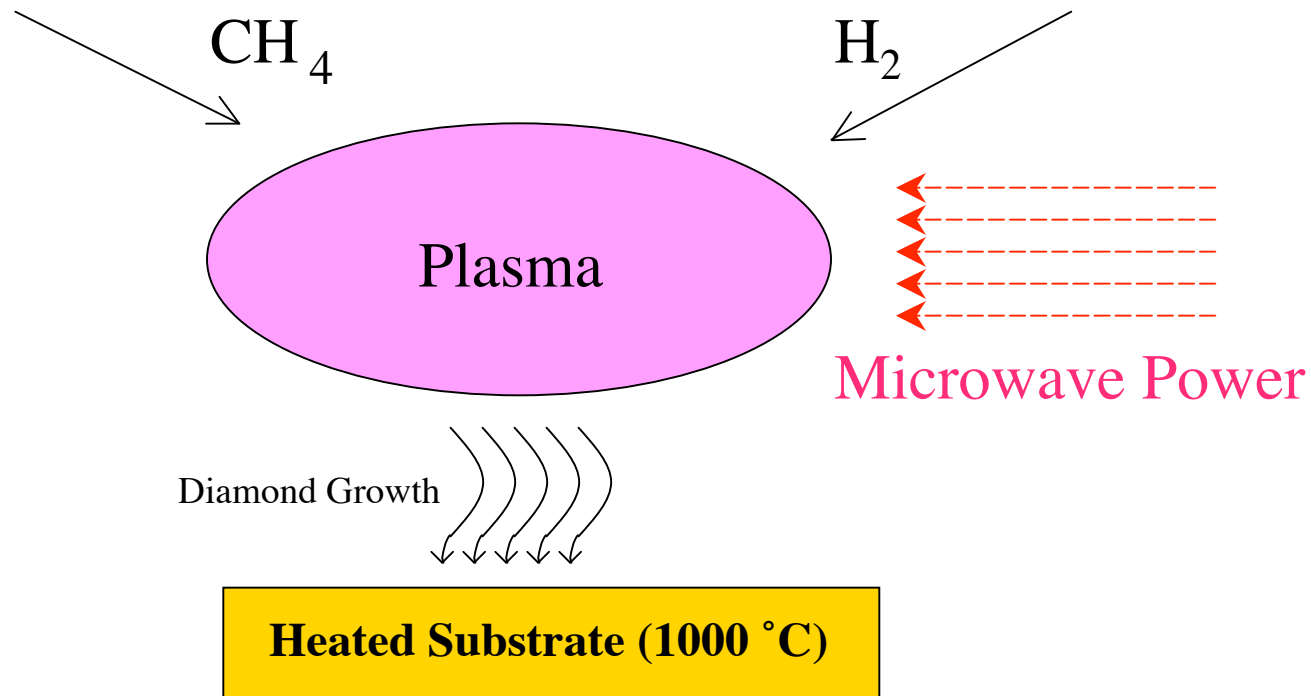


- Feature linewidths down to about 5 μm .
- Probes are made of tungsten 0.5 μm thick.
- “Lift-off” process used



Microwave Plasma Chemical Vapor Deposition (MPCVD)

(Prof. Yogesh Vohra, Univ. of Alabama)

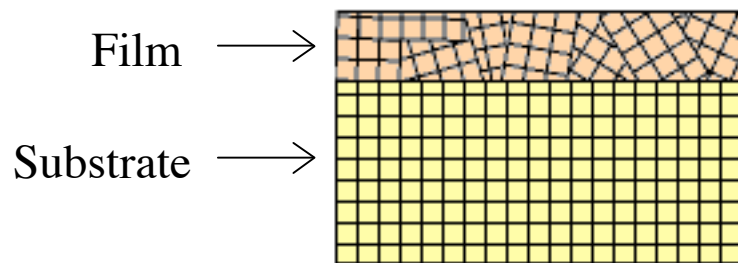


- 2% methane and 98% hydrogen gas mixture
- Plasma generated by a 1.2 kW magnetron operating at 2.45 GHz
- ***Epitaxial*** diamond onto diamond substrates
at a growth rate of about 10 $\mu\text{m}/\text{hour}$

Epitaxial Diamond Growth onto the Anvils

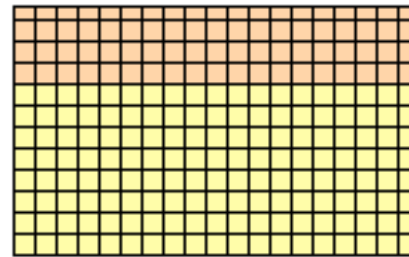
Epitaxial Growth: Growth of single-crystal film on a single-crystal substrate, with the crystallographic orientation of the film matching that of the substrate.

Non-Epitaxial Growth



- film and substrate remain distinct from one another, with a clear, identifiable boundary between them.

Epitaxial Growth



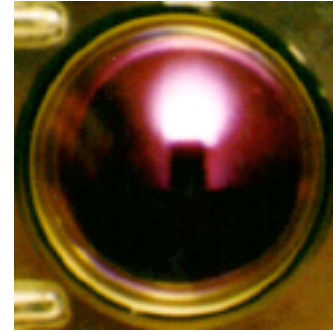
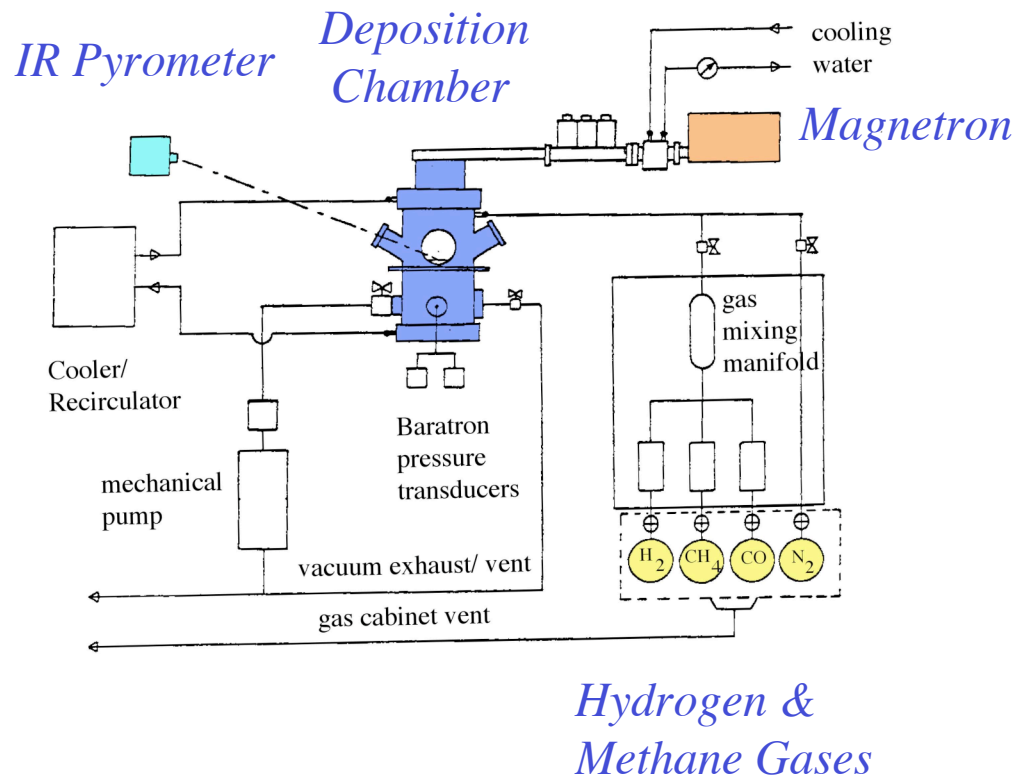
- film and substrate merge into one: Essentially, no boundary (or an “invisible” boundary) between them

→ Properly fabricated designer anvils appear to be as strong/robust as standard diamond anvils. Designer Anvils have been used up to 2.8 Mbars

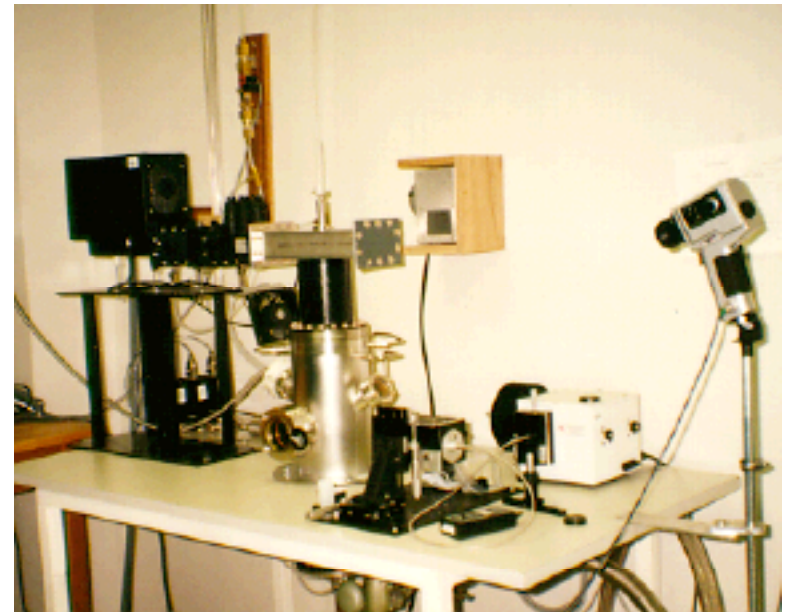
CVD Diamond Deposition System at the University of Alabama

(Prof. Y.K. Vohra, Dept. of Physics)

Schematic of CVD Reactor System



Microwave Plasma during CVD

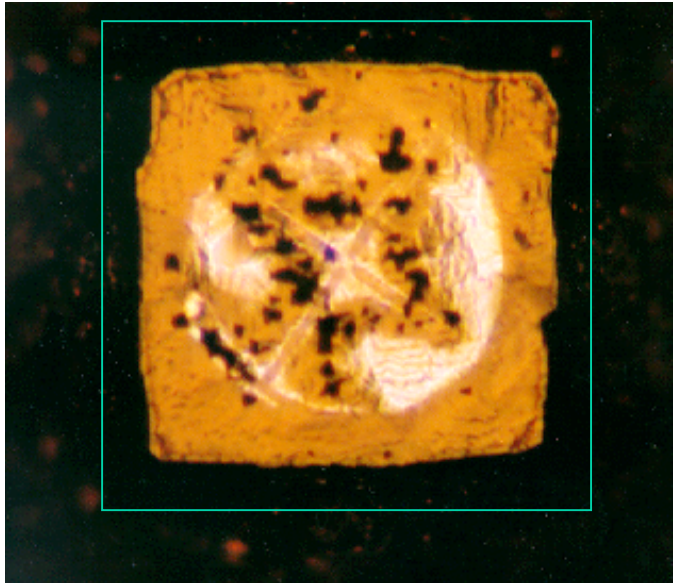


Univ. of Alabama MPCVD System

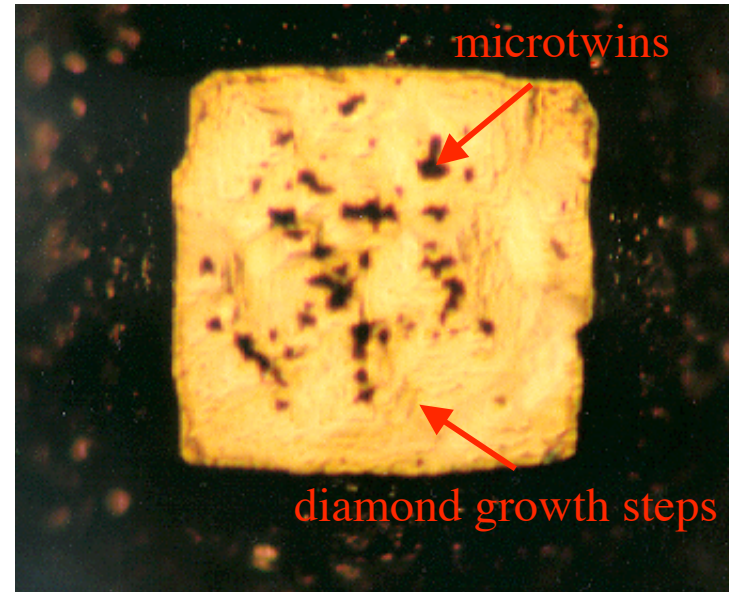
CVD Synthetic Diamond Films are of Very High Quality

As-Grown CVD Diamond Film on a (100)-Oriented Diamond Anvil

Transmitted Light

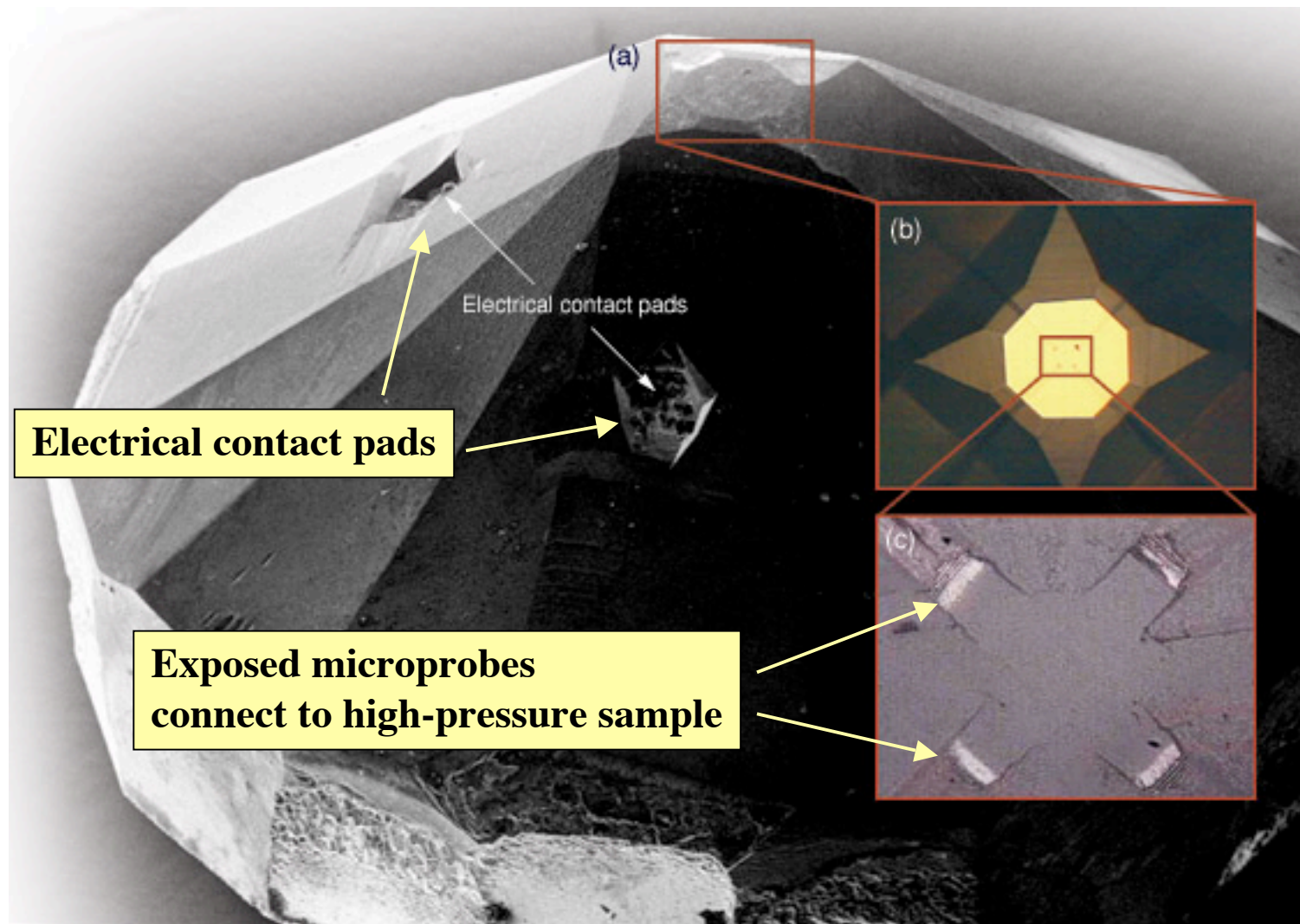


Reflected Light

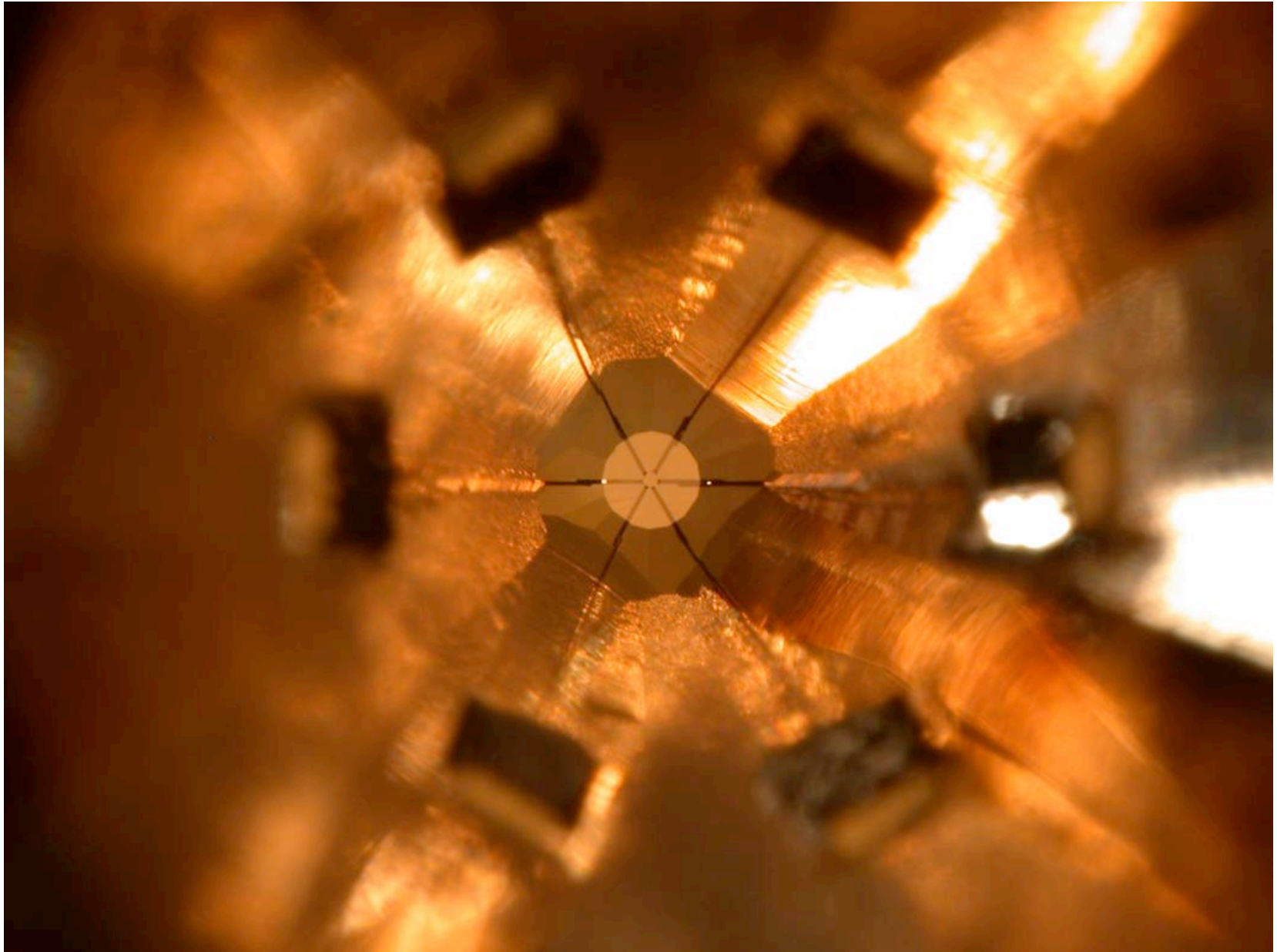


- 20 μm of CVD diamond
- high-quality diamond film (small sp^2 -carbon content)
- Zr microprobe test pattern fabricated

Completed Designer Diamond Anvil



A “Designer” Diamond Anvil

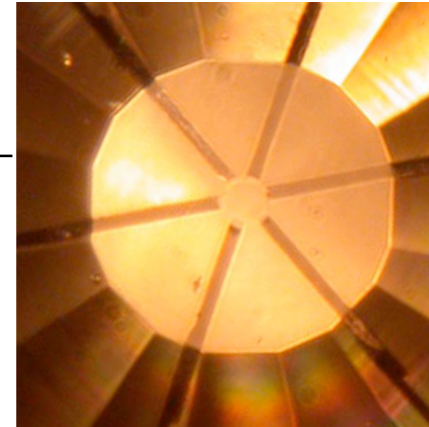


Wide Range of Possible Experiments

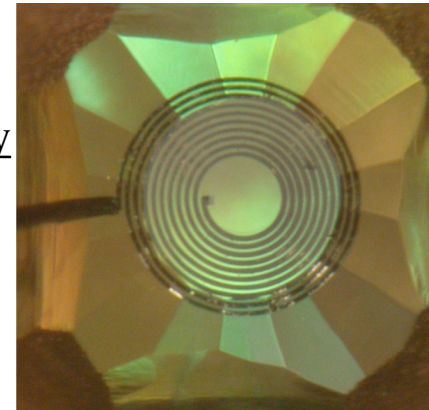


Designer Diamond Anvil

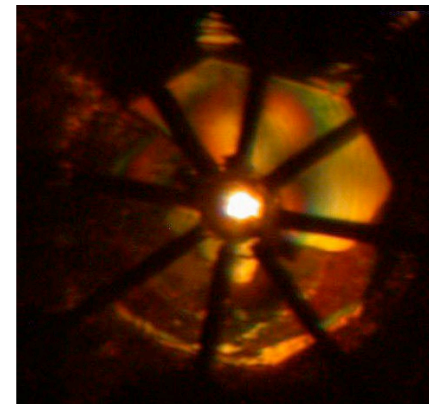
Electrical Conductivity
Experiments



Magnetic Susceptibility
Experiments



High-P/High-T
Experiments



*Many other types of high-pressure
experiments yet to be explored ?*

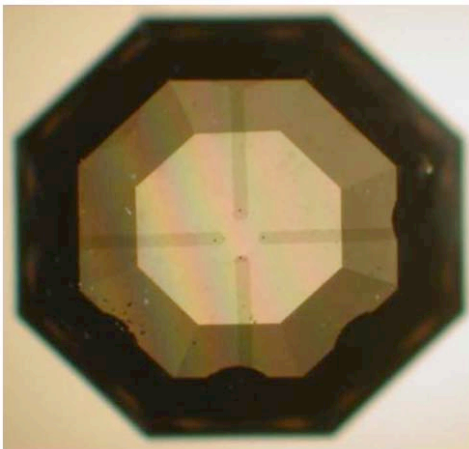
Conductive Paths in Diamond by Boron-Implantation

(iDAC, “intelligent” diamond anvils)

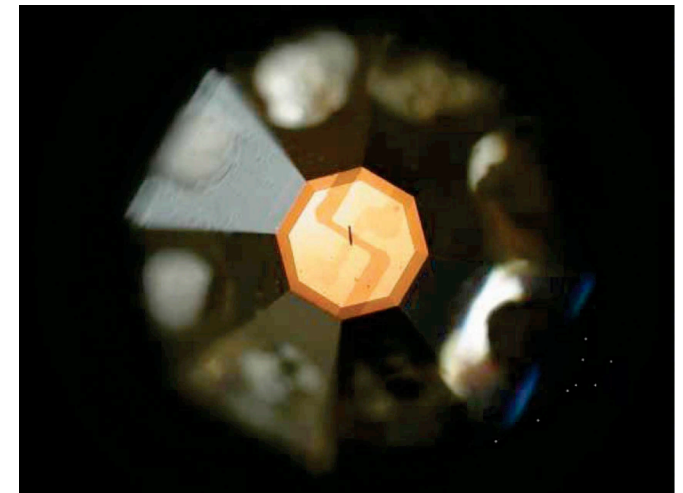
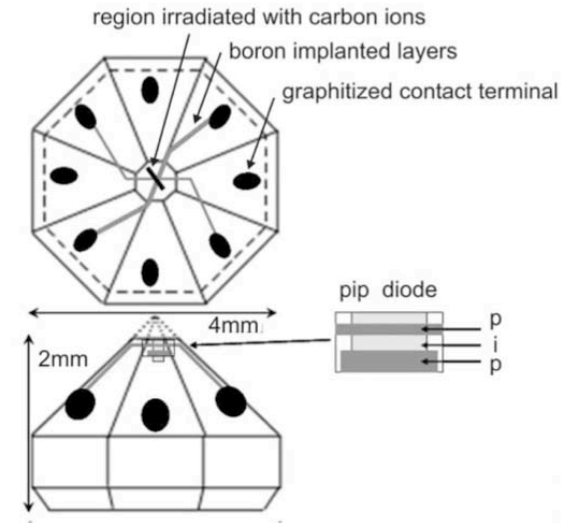
H. Bureau, et. al., *High Pressure Research* **26**, 251 (2006).

- High-energy ion implantation of boron atoms
- implantation doses $> 10^{16}$ boron atoms per cm^2
(> 1 boron atom per \AA^2)
- ions are implanted up to $3\text{ }\mu\text{m}$ below diamond surface
- diamond temperature $> 800\text{ }^\circ\text{C}$ to avoid graphitization

iDAC Conductivity Anvil



Pressure or Temperature Sensor Anvil

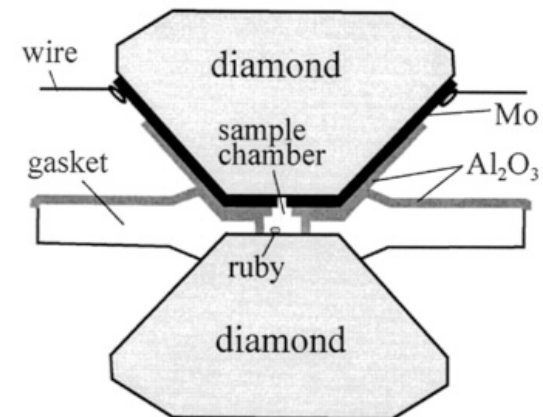
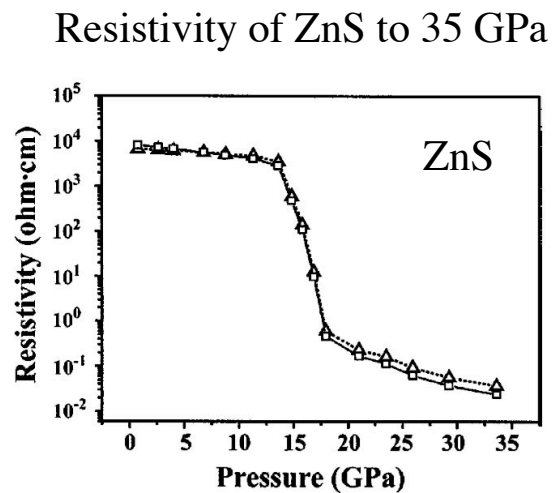
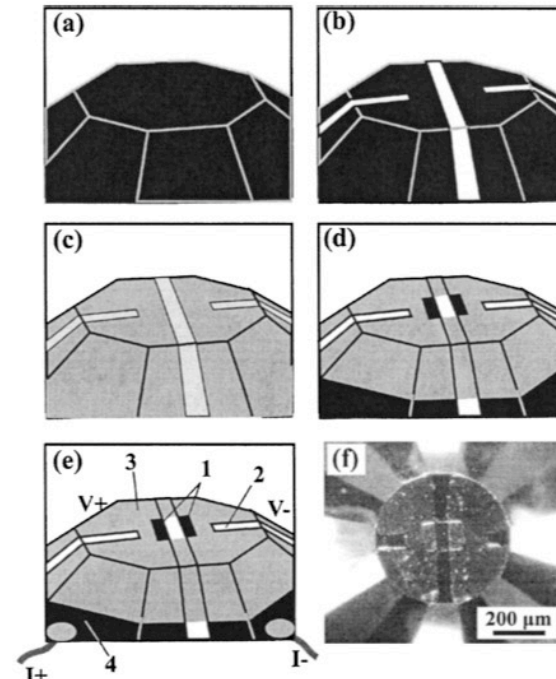


H. Bureau, et. al., *High Pressure Research* **26**, 251 (2006).

Another Diamond Anvil Integrated Circuit Technique

- Y. Han, C. Gao, et. al., *Appl. Phys. Lett.* **86**, 64104 (2005).

- Molybdenum used as electrical conductor
- Alumina (Al_2O_3) deposited for electrical insulation
- Photolithographic patterning used
- Tested up to 106 GPa



Y. Han, et. al., *Appl. Phys. Lett.* **86**, 64104 (2005).

Focused Ion Beam (FIB) for Deposition of Metal

Characteristic Parameters:

- Gallium ion (Ga^+) beam
- Typical energy $\approx 30\text{-}50\text{ keV}$
- Beam current $\approx 1\text{-}20\text{ nA}$
- Imaging resolution down to $5\text{-}7\text{ nm}$
- Chamber pressure $\approx 10^{-7}\text{ mbar}$

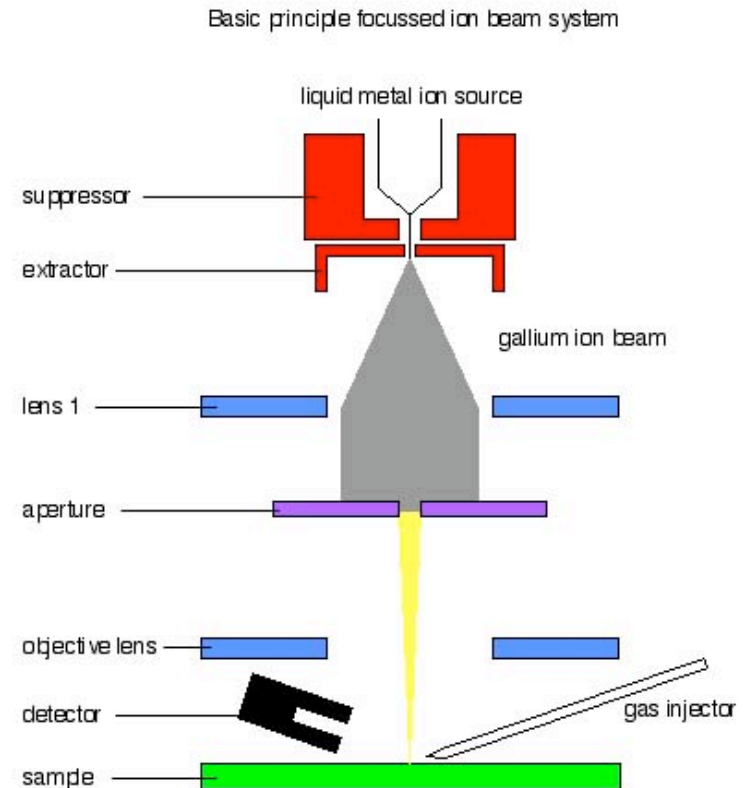
Very versatile device! Uses:

- High Magnification Imaging
- Precision Ion Milling/ Sectioning
- **Deposition of Metals (e.g., Pt and W)**



Ion-assisted chemical vapor deposition (IACVD)

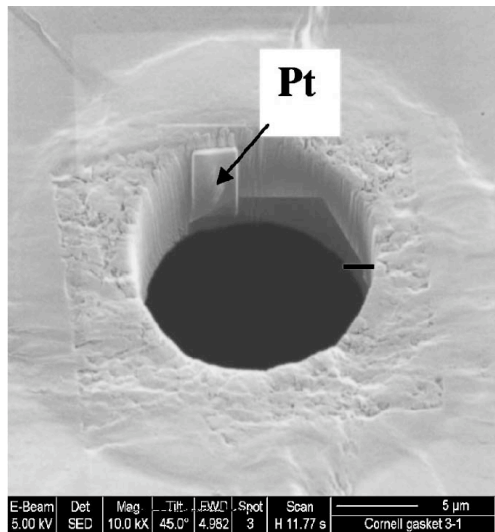
- organometallic vapor introduced into chamber ($\text{C}_9\text{H}_{16}\text{Pt}$) and adsorbed onto the substrate
- adsorbed layer is decomposed by the ion beam, depositing a thin metal layer.



Focused Ion Beam (FIB) for Deposition of Metal

Pt X-Ray Marker in Gasket Hole

- Pt is $2.8 \times 4 \times 0.5 \mu\text{m}$



A.L. Ruoff, et. al., *Rev. Sci. Instrum.* **76**, 36102 (2005).

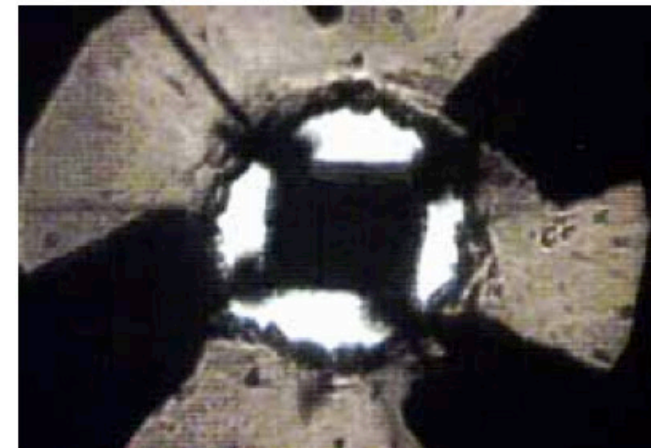
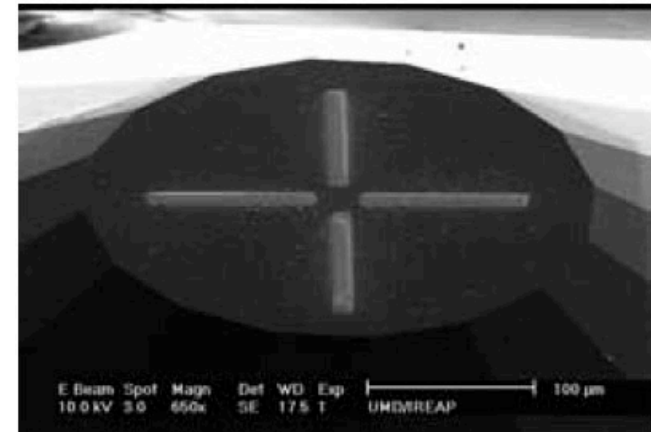
Advantages:

- combines deposition *and* patterning into the same step.
- also gives milling/sectioning capability into diamond?

Possible Disadvantages/ Questions:

- Expensive and difficult to get access (at least at LLNL)
- Unknown how well Pt adheres to diamond

Metal Probes on a Diamond Anvil



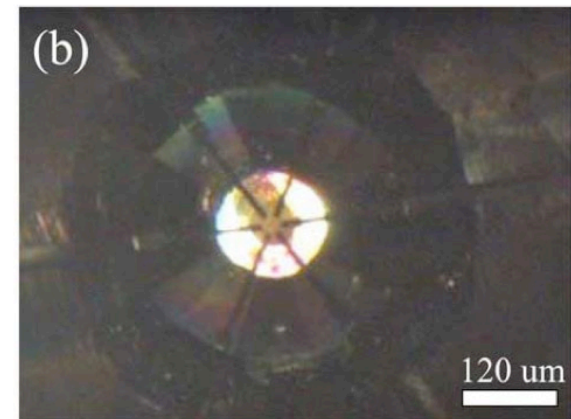
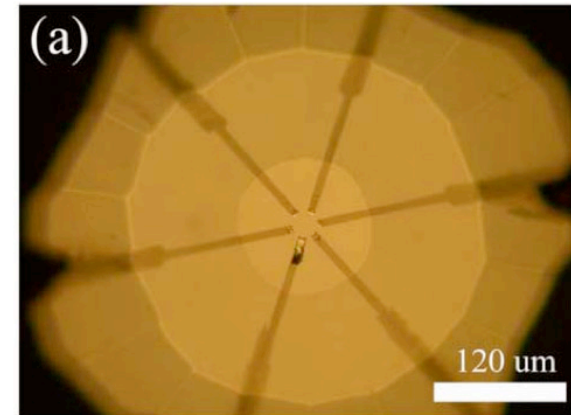
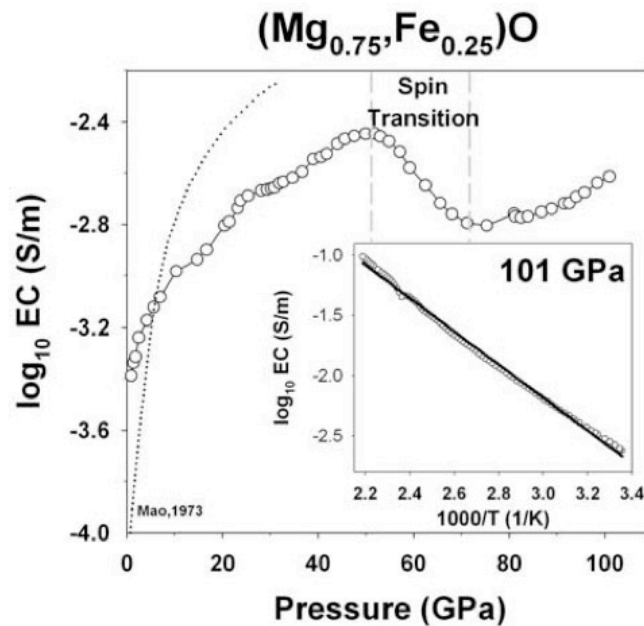
Z-X Shen, T. Cuk, et. al., in *CDAC (Carnegie/DOE Alliance Center) 2006 Annual Report*

Example: Conductivity of Ferroperricite ($\text{Mg}_{0.75}\text{Fe}_{0.25}\text{O}$)

J-F Lin, et. al., *Geophys. Res. Lett.* **34**, L16305 (2007)

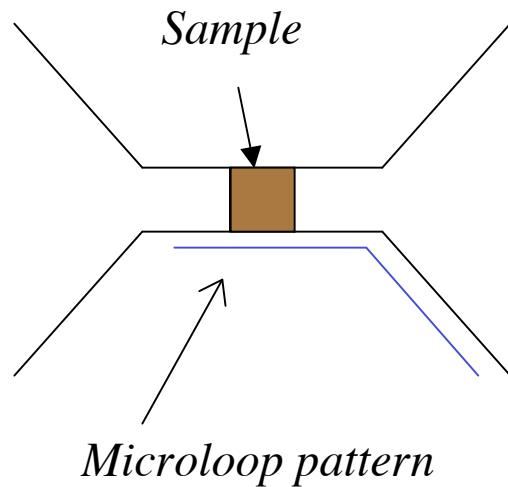
Ferroperricite: Constituent of the Earth's lower mantle

- 6-probe designer anvil
- 120 μm diamond flat, 60 μm diameter sample



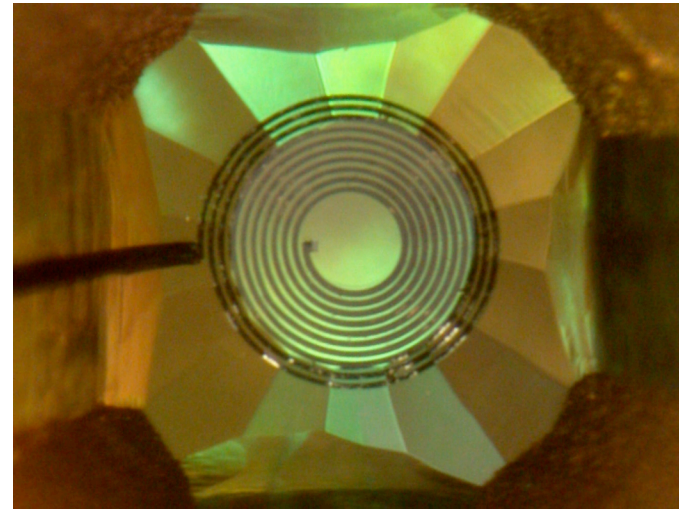
High-Pressure Magnetic Susceptibility Experiments

Designer Anvils with diamond-embedded microloops:



- Mechanically robust
- Very high signal-to-background

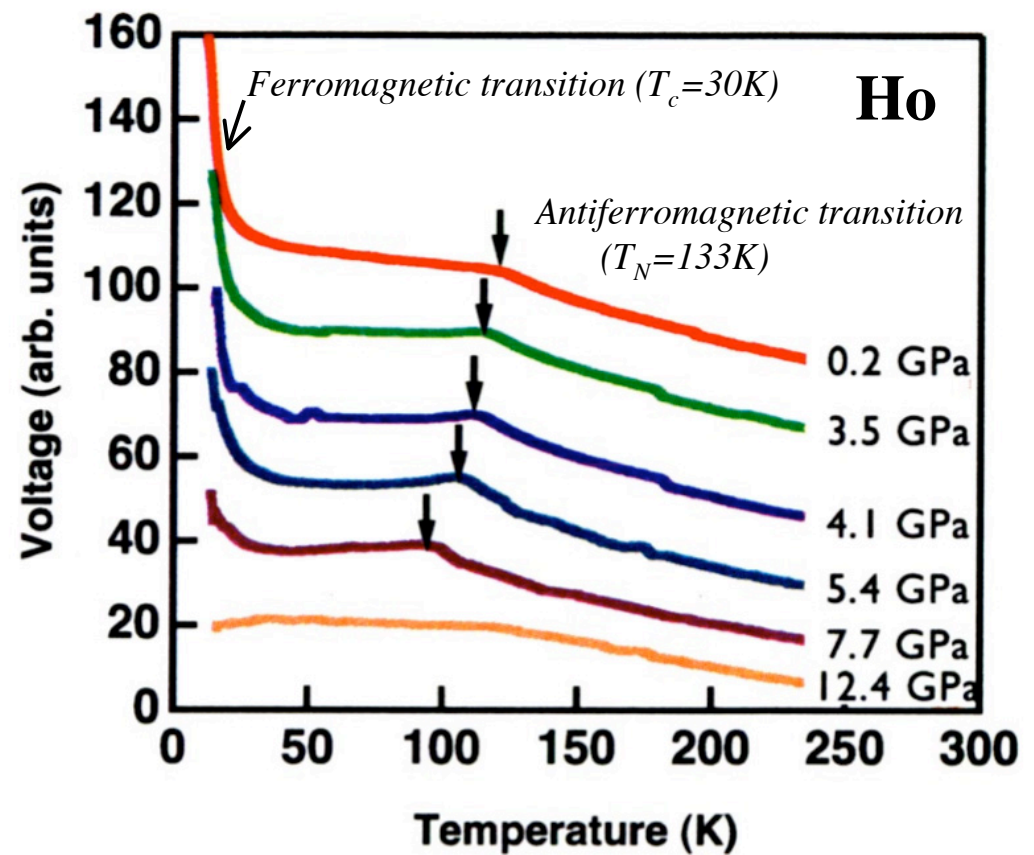
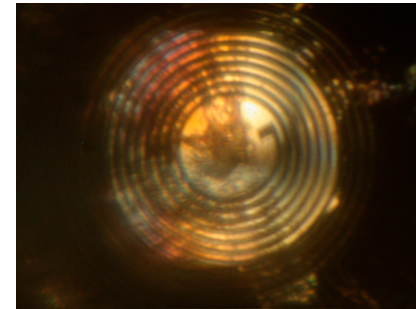
Multiloop Designer Anvil



- 10-turn magnetic sensing loop on diamond culet.
- lines are 5 μm wide and 0.5 μm thick.

Example: Holmium Magnetic Susceptibility

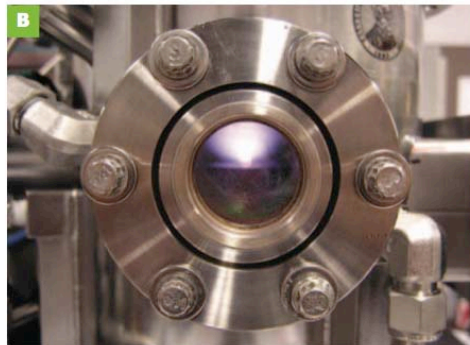
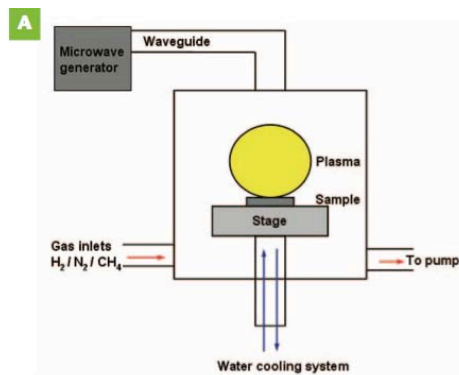
- Holmium sample ($\approx 75 \mu\text{m}$ diameter)
- Ho undergoes two magnetic transitions involving f-electron magnetic ordering
- Able to track both the ferromagnetic transition and the antiferromagnetic transition



The Future: “Giant” Diamond Anvils ?

Can the diamond CVD process be scaled up to create ultra-large diamond crystals?

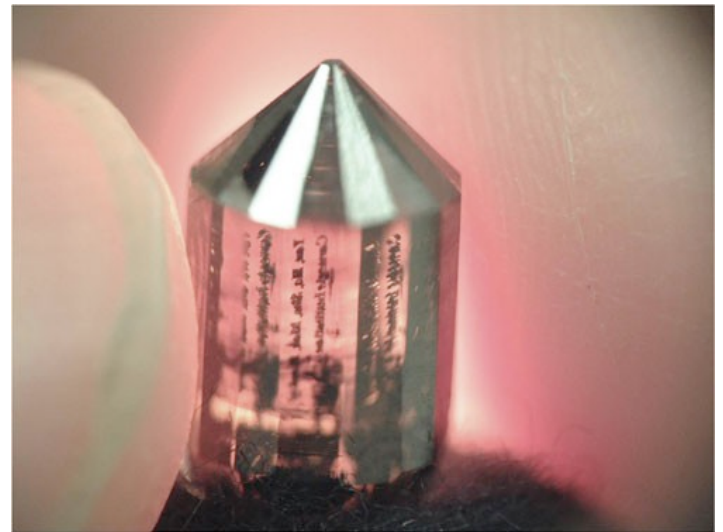
- Microwave-plasma CVD process ($\text{H}_2/\text{CH}_4/\text{N}_2$)
- growth rates of $\approx 100 \mu\text{m/hr}$



CVD-grown 5 carat diamond

(after cutting and polishing a 10 carat stone)

R.J. Hemley, C-S Yan, et. al., Carnegie Institution
(www.sciencedaily.com)



Summary:

- A complete set of diamond-based fabrication tools now exists for making a wide range of different types of diamond anvils which are tailored for various high-P applications.

- Current tools include:

- CVD diamond deposition (making diamond)
- Diamond polishing, laser drilling, plasma etching (removal of diamond)
- Lithography, 3D laser pantography (patterning features onto diamond)
- Metal deposition (putting electrical circuits and metal masks onto diamond)

- Current applications include the following:

- Electrical Conductivity
- Magnetic Susceptibility
- High-P/High-T

- Future applications may include:

- NMR
- Hall Effect
- de Haas - Shubnikov (Fermi surface topology)
- Calorimetry
- thermal conductivity
- *Many others ?*

